Impact of a Surface Sealant Application on the Color Stability of a Nano-hybrid Composite Resin

Yüzey Örtücü Uygulanmasının Nano-hibrit Kompozit Rezinin Renk Stabilitesi Üzerindeki Etkisi

**ORIGINAL ARTICLE**

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**ABSTRACT**

Objective: Mouthwashes containing chlorhexidine can cause discoloration in restorative materials when used for the long-term. To prevent staining of a restorative material, several protective materials are used. The aim of this *in vitro* study was to evaluate the impact of a surface sealant on the color stability of a nano-hybrid composite resin material immersed in different mouthwashes.

Methods: A total of 42 composite resin discs (10x2 mm) were prepared with a nano-hybrid composite resin (Herculite XRV Ultra) using Teflon moulds. The composite specimens were first divided into two groups (with or without a surface sealant) then into three subgroups (n=7) according to the mouthwashes (Gengigel, Oderol, and Chlorhex). After SS application to 21 specimens, all the discs were subjected to baseline color measurements using a spectrophotometer according to the Commission Internationale de l’Eclairage L*a*b* system. The specimens underwent thermal cycling. Following immersion in different mouthwashes for 24 hours, the color measurements were repeated. The ∆E data were assessed using Kruskal-Wallis one-way ANOVA. Mann-Whitney U test was performed for different immersion mouthwashes (p<0.05).

Results: Statistically significant differences (p<0.05) were found between the unsealed composite specimens immersed in Chlorhex and the other mouthwashes in terms of ∆E values. The Chlorhex

**ÖZ**


Yöntemler: Bir nanohibrit kompozit rezin materyali (Herculite XRV Ultra) kullanılarak 42 adet disk şeklinde kompozit örnekleri (10x2 mm) hazırlanı. Örnekler önce iki grubu (yüzey örtücü uygulanan veya uygulanmayan) ardından dallanılan ağız gargaları (Gengigel, Oderol ve Chlorhex) göre üç alt gruba (n=7) ayrıldı. Yırdı bir kompozit örneğine yüzey örtücü uygulamasını takiben, tüm disklerin başlangıç renk ölçümlerini bir spектрофотометre (Vita Easyshade) kullanarak Uluslararası Aydınlatma Komisyonu L*a*b* sisteminde göze çarpırdı. Örneklere termal siklus uygulandı. Yırdı dört saat boyunca farklı ağız gargalarında bekletilen örneklerin renk ölçümleri tekrarlandı. ∆E verileri Kruskal-Wallis tek yönlü one-way ANOVA kullanarak değerlendirildi. Farklı gargalar için Mann-Whitney U testi kullanıldı (p<0.05).

Bulgular: Yüzey örtücü uygulanmamış kompozit örneklerinde, Chlorhex ve diğer gargalar arasında ∆E değerleri bakımından

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Introduction

A coordinate interaction can occur between the ultimate aesthetic characteristics of dental restoration and the efficacy of the finishing and polishing strategies applied (1). The utilization of solvent polishing liquids with unique systems has been suggested alongside the immediate placement and finishing of resin-based restorative materials. Such an application has been recommended to overcome the various defects feasibly related to the physical characters of the materials as well as the potential faults of the operator (2). The postoperative sealing agents are generally described as rebonding/glazing/liquid-polishing agents or surface sealants (SSs) (2,3). These SSs are placed on the surface of the restoration and the neighbouring tooth structure, thereby boosting the marginal sealing, and also penetrating any surface micro defects and/or gaps formed during the material placement and polymerization (2,3).

The color and surface roughness of the teeth are critical qualities in terms of aesthetics when designing a smile (4). Since the development of composite materials in 1960, attempts have been made to boost the durability of composite restorations. However, as relatively little progress has been made to date, the visual characteristics of these materials still need to be upgraded (5). The color alteration experienced by composites is multifactorial, being related to the intrinsic staining and extrinsic discoloration of the material. The intrinsic factors are related to changes in the chemical balance of the resin matrix and the matrix/particle interface, while the extrinsic factors are associated with the consumption of coloring liquids from exogenous sources related to hygiene, nourishment, and smoking (6).

In recent decades, the number of individuals who use mouthwashes for anti-microbial control has grown, not only due to professional guidance but also due to the capability of such liquids to provide a cooling sensation and to diminish halitosis (7). Mouthwashes have diverse ingredients including alcohol, emulsifiers, organic acids, detergents, and dyes. It should be noted that composite resins exposed to ethanol display decreased microhardness values when compared with non-exposed materials (8). Alcohol appears to act as a plasticizer of the polymeric matrix, thereby making the resin material more ductile (9). Additionally, ethanol can reduce the adhesion between the resin matrix and inorganic fillers, which may increase the softening of the surface and leads to discoloration of the resin material (8).

Endeavors to quantitatively characterize the color alteration experienced by a tooth-colored restoration have been facilitated by the Commission Internationale de l'Eclairage (CIE), which has presented L*a*b* three-dimensional color estimation system whereby the L* axis assesses the luminosity or lightness (extending from 0 [black] to 100 [white]), the a* coordinate assesses the redness (a>0) or greenness (a<0), whereas the b* coordinate assesses the yellowness (b>0) or blueness (b<0) (10,11). The quantitative estimation of the entire alteration in color (∆E) hence consolidates a unique equation for a qualitative application relating to both discernment and acceptance limits (10,11).

In dental practice, mouthwashes may need to be used for a long time if the patient is to benefit from both their wound-healing properties and their antibacterial properties. However, chlorhexidine (CHX) can result in a permanent color change following long-term use. Besides, reversible local side effects such as discoloration of teeth, and the tongue, impaired taste sensation (12), increased formation of supragingival calculus and mucous membrane irritation, and desquamation (13) are associated with the use of CHX mouth rinse in long-term use. Oderol mouthwash contains zinc lactate and CHX, although the manufacturer claims that it does not cause discoloration after long-term use. Gengigel, which is a hyaluronic acid mouthwash, is marketed offering a wound-healing potential. To the best of our knowledge, no studies have yet examined the discoloration associated with both Oderol and Gengigel in composite restorations following long-term use.

The first aim of this study was to evaluate the long-term use of three different types of mouthwash on the color stability of composite restorations. The second aim of this study was to assess the effect of SS application on the coloration resistance of a nanohybrid resin composite soaked in different types of mouthwash. The null hypothesis was that the application of a SS would not be able to protect the color stability of a nano-hybrid composite restoration after using different mouthwashes in the long-term.

Keywords: Color change, mouthwash, surface sealant, composite resin, thermal cycling

Conclusion: The application of a low viscosity liquid surface sealant material did not show the expected effect on the color stability of a nano-hybrid composite resin in terms of three different mouthwashes.

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Method
Sample Preparation
Forty-two disc-shaped specimens (10.0 mm inner diameter x 2.0 mm thickness) were prepared in Teflon moulds utilizing the Herculite XRV Ultra (Kerr Corporation, Orange, CA, USA) shade A2 nanohybrid composite material. Each material was covered with transparent polyethylene matrix strips (Kerr Hawe Striproll, Kerr Corporation, Orange, CA, USA) on each side to ensure a uniform finish. The composite resins were compressed between two microscopic glass slides to expel any excess material. Photopolymerization was performed for 20 seconds using a VALO Cordless (Ultradent Products Inc., South Jordan, USA) light-emitting diode (LED) device with an irradiance of 1,800 mW/cm². The directly lighted surfaces were progressively polished using aluminium oxide polishing discs (Sof-Lex, 3M ESPE, St Paul, MN, USA). A single operator performed the polishing process with a low-speed handpiece at around 4,000-5,000 rpm. Each disc was used on a surface for 15 s. Then, the polished surfaces were water-rinsed for 60 s to expel any surface debris. Afterward, they were air-dried for 30 s. The thickness of each sample was checked using a digital calliper (Yamer, Izmir, Turkey). To guarantee the absence of any deformities, the surfaces were inspected under a metallographic microscope (ME 600 Eclipse, Nikon-Kogaku, Tokyo, Japan). Then, the specimens were submersed in artificial saliva (14) (1 L aqua, 0.1 g H₂BO₃, 2.4 g KCl, 1.7 g NaCl, 0.1 g MgCl₂·6H₂O, 0.2 g CaCl₂·2H₂O, 0.2 g KSCN, 0.7 g KH₂PO₄) and stored in an incubator (Memmert GmbH, Schwabach, Germany) at a temperature of 37±1 °C for 24 h to provide complete polymerization.

Twenty-one of the specimens were etched with 37% phosphoric acid (Etchant Gel; Prime Dent, Chicago, IL, USA) for 15 s, then washed with distilled water and air-dried. Later, the BisCover LV SS was applied directly using a micro brush for 15 s. The top surfaces of the specimens were tenderly air-thinned for 3 s and then light-cured for 30 s, as recommended in the manufacturer’s instructions, using a LED light unit. The other twenty-one specimens remained unsealed. The compositions of all the materials used in this study were detailed in Table 1. The specimens were stored in artificial saliva at 37±1 °C for 24 h to provide complete polymerization of the SS. At that point, all the specimens were subjected to ageing through 1,500 thermal cycles (SD Mechatronik GmbH, Feldkirchen-Westerham, Germany) at temperatures rotating between 5 °C and 55 °C with a stay time of 30 s in each water bath (15).

After the thermocycling, the specimens were divided into three groups (n=14) based on the mouthwash in which they would be immersed (Table 1): Group 1 - Gengigel, Group 2 - Oderol, and Group 3 - Chlorhex. The specimens were stored in 20 mL of each mouthwash for 24 h, which was equivalent to 2 min/day of mouthwash use for two years (16). The specimens within the mouthwashes were kept at 37 °C throughout the experiment, and they were shaken every 3 h to ensure their homogeneity (17). At the end of the experiment, the specimens were evacuated and then submerged in distilled water.

Color Measurements
Following the application of the surface sealant to 21 composite specimens, the baseline color measurement of all the specimens was performed under a white background (Gardner Laboratory Inc., Bethesda, MD, USA) using a spectrophotometer (Vita Easyshade, Vita Zahnfabrik, Bad Säckingen, Germany). This device is specifically designed for colour measurement. Before the measurement, the top surface of each sample was dried using tissue paper and the contact guide of the spectrophotometer was situated at the center of the sample’s surface. Three successive readings were taken for each sample and then averaged.

After thermal cycling and immersion of all 42 composite specimens in the mouthwashes for 24 h, the color measurements were repeated as detailed above. Based on the L*, a*, and b* values, the overall color change (ΔE) was calculated using the following equation:

\[
ΔE = \left[ (ΔL^*)^2 + (Δa^*)^2 + (Δb^*)^2 \right]^{1/2} \quad (18).
\]

ΔE values ≥3.3 were considered to be clinically improper (19).

Statistical Analysis
The distribution of the data was determined using the Shapiro-Wilk test.

<table>
<thead>
<tr>
<th>Table 1. Materials used in the study</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Material</strong></td>
</tr>
<tr>
<td>Herculite XRV Ultra (A2)</td>
</tr>
<tr>
<td>(Nano-hybrid composite resin)</td>
</tr>
<tr>
<td>BisCover™ LV</td>
</tr>
<tr>
<td>(Low viscosity liquid polish)</td>
</tr>
<tr>
<td>Gengigel (Hyaluronic acid mouthwash)</td>
</tr>
<tr>
<td>Oderol (Zinc lactate mouthwash)</td>
</tr>
<tr>
<td>Chlorhex (Chlorhexidine gluconate mouthwash)</td>
</tr>
</tbody>
</table>
Due to the non-normal distribution of the data, the differences in the color changes (ΔL*, Δa*, Δb* and ΔE) between the different mouthwash groups were analyzed using a Kruskal-Wallis one-way ANOVA. To assess the differences between the sealed and unsealed groups, a Mann-Whitney U test was performed. Data were submitted for statistical analysis at the p=0.05 level of significance. The statistical calculations were performed using SPSS for Windows 24.0 (SPSS Inc., Chicago, IL, USA).

**Results**

The mean, standard deviation, and median (minimum-maximum) of the ∆E, as well as of the ∆L, ∆a, and ∆b values, for the sealed and unsealed groups following immersion in different mouthwashes, are presented in Tables 2 and 3.

Statistically significant differences were found between ∆E values of the specimens without surface sealant but immersed in Chlorhex and the other mouthwashes tested. Chlorhex showed a greater degree of color alteration (p<0.05), followed by Gengigel and Oderol (Table 2).

Regarding the SS, statistically significant differences (p<0.05) were found between the groups in relation to the ∆a values (sealed and unsealed), except for Oderol and Chlorhex groups (Table 3).

Concerning the tested mouthwash solutions, no statistically significant differences were found between the groups (p>0.05) (in relation to the ∆L, ∆a, and ∆b values), except for the ∆L values in the Chlorhex group (p<0.05) (Table 3).

**Discussion**

The null hypothesis was partially accepted in this study. Although there was a considerable increase in ∆E values after the SS application (except for Chlorhex group), no statistically significant difference in color was observed between the sealed and unsealed composite specimens (p>0.05). The preservation of the color throughout the functional period of dental restorations is one of the foremost aesthetic characteristic considerations concerning composite materials. However, this characteristic is not consistent among the different available restorative materials. In our study, we used a nano-hybrid composite material that could be used both in the anterior and posterior restorations, due to the superior polishability, better optical and aesthetic properties, and less polymerization shrinkage (20). Chairside polishing of composite resins with sealant agents should provide improved stain resistance. However, the success and longevity of these sealant agents on composite resin restorations are still unknown. Since one of our purposes in the study was to evaluate the effect of a surface sealant on the color stability, we preferred to choose only one resin composite material.

**Table 2. ΔE median (min-max) and mean ± standard deviation of the experimental groups**

<table>
<thead>
<tr>
<th>Without sealant</th>
<th>With sealant</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Mean ± SD</strong></td>
<td><strong>Median (min/max)</strong></td>
</tr>
<tr>
<td>Gengigel</td>
<td>2.21±1.46&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td>Oderol</td>
<td>1.94±1.75&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td>Chlorhex</td>
<td>5.14±0.83&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td>p</td>
<td>0.005</td>
</tr>
</tbody>
</table>

The different lowercase letters in the columns indicate statistically significant differences between the mouthwash groups (p<0.05), while the uppercase letters in the lines indicate statistically significant differences between the sealed and unsealed groups (p<0.05).

**Table 3. ΔL, Δa, Δb median (min-max) and mean ± standard deviation of the experimental groups**

<table>
<thead>
<tr>
<th>Without sealant</th>
<th>With sealant</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Median (min/max)</strong></td>
<td><strong>Median (min/max)</strong></td>
</tr>
<tr>
<td>Gengigel</td>
<td>ΔL -0.7 (-0.4/0.1)&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>Δa 0.3 (-0.1/0.4)&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>Δb -0.8 (-3/-0.6)&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td>Oderol</td>
<td>ΔL 0.3 (-5.6/1)&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>Δa 0.1 (-1/0.5)&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>Δb -0.9 (-2.3/0.2)&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td>Chlorhex</td>
<td>ΔL -4.9 (-6.7/-4.2)&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>Δa 0.1 (-0.2/0.2)&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td></td>
<td>Δb -0.8 (-2.1/-0.2)&lt;sup&gt;ab&lt;/sup&gt;</td>
</tr>
<tr>
<td>p</td>
<td>&lt;0.05</td>
</tr>
</tbody>
</table>

The different lowercase letters in the columns indicate statistically significant differences between the mouthwash groups (p<0.05), while the uppercase letters in the lines indicate statistically significant differences between the sealed and unsealed groups (p<0.05).

min: Minimum, max: Maximum
In most prior studies, a spectrophotometer was used in accordance with the CIE’s L*a*b* framework to measure the ∆E values (21,22). This electronic device uses a normal light in a dark component, separated from the surrounding light, to measure the color and, thus, the results are convincing, definitive, and quotable. The ∆E value of composite resins that is greater than 3.3 indicates a clinically critical and unacceptable color change (18).

The color stability of aesthetic composite restorations is influenced by several extrinsic and intrinsic variables. Extrinsic discoloration results from factors such as poor oral hygiene, dietary, and smoking patterns. The use of mouthwash is another extrinsic variable that influences the color stability of aesthetic composite restorations. Yet, the use of mouthwash is also known to help control caries and periodontal illnesses (23,24).

Therefore, in our study, we evaluated the staining effect of two different mouthwashes with very high periodontal effectiveness, containing different concentrations of CHX gluconate as well as a mouthwash containing hyaluronic acid.

In the present study, all mouthwashes caused a color alteration on the composite resins. The Chlorhex with a ∆E value of 5.22 was found as the mouthwash that caused the greatest degree of color alteration among all the tested mouthwashes (Table 2). This high value has been characterized as unacceptable for in vitro conditions (25). The absorption of the alcohol particles contained in mouthwashes into the resin matrix may result in the softening of the composite resin’s surface and so contribute to its discoloration, which may explain the outcomes observed for Chlorhex (26). One of the mouthwashes tested in this study was Oderol. It incorporates zinc, which is an essential element in plaque, saliva, and enamel. Zinc is added to oral healthcare products to control plaque and to prevent calculus formation. In the presence of plaque and saliva, its permanence may persist for hours in the oral environment (27). According to our findings, the specimens immersed in Oderol, which contained a lower amount of CHX gluconate (0.025 mg) than Chlorhex, did not show unacceptable levels of color change. This acceptable color change may be due to the concentration of CHX gluconate mouthwash ranging from 0.2% to 0.12% (28).

The mechanism of staining by CHX gluconate is not known exactly. In the literature, three possible mechanisms were reported (29). 1) CHX may increase the non-enzymatic browning reaction of protein and carbohydrate in the acquired pellicle; 2) CHX denatures components within the dental pellicle, accelerate the formation of pigmented sulfides of iron; 3) CHX precipitates dietary chromagens. According to our results, Chlorhex with a higher concentration of CHX gluconate caused the most severe color alteration of all mouthwashes. The use of CHX gluconate mouthrinse has been reported to cause staining in earlier studies (28,30,31). From this perspective, this study carries confirmatory value.

The use of an SS, which comprises BIS-GMA, UDMA, and TEG-DMA without filler particles, may assist with removing or minimizing the color variations seen in relation to composite resins. Such materials are characterized by low thickness and high usefulness (32). The use of a SS serves to fill any microstructural defects of the surface and so decreases the surface roughness and the presence of bubbles. Different opinions exist as to the SS, which can alter the surface properties, color stability, marginal microleakage, and optical properties of composite resins (33,34).

There are contradictions among the results of previous studies in this area (24,33,35). According to Doray et al. (33), SS can promote the resistance of temporary composite resin immersed in red wine, coffee, and cranberry juice to staining. However, Soares et al. (35) applied a reflectance analysis and showed that the coffee-related surface coloring was higher in the groups that applied SS. Similar to the results of the present study, Lee and Powers (24) stated that the color alterations seen in the sealed group subjected to staining by CHX, tea, and mucin were not notably different when compared to those seen in the unsealed group. Differences in the chemistry, polymerization approaches, and thicknesses of the sealants may impact their sensitivities to coloring. Regarding the current study, an unexpected phenomenon occurred after the polymerization of SSs on the surfaces of the composite samples. Although the initial appearance was a glossy surface, air bubbles formed in the sealant material during the application with a brush, might provide a suitable environment for the discoloration of the material, as they created irregular areas on the restoration surface after polymerization. The variations between the ∆E values of the tested specimens in the present study may also be attributed to the long-term immersion in the mouthwashes, which destroys the integrity of the SS layer. This destroying could lead to microcrack formations and the removal of nonadherent surface particles. The increased thickness of the sealant agent may be responsible for the various degrees of discoloration and deterioration of the surface sealant (24). Based on these, it can be said that the SS application may have no positive effect (33).

The discoloration of composite resins can be considered in relation to their degree of water absorption and the hydrophilicity of the resin matrix (36). If the resin matrix can retain water, it is also able to retain any other liquid, which eventually leads to discoloration. Extraordinary water absorption induces the extension and plasticization of the composite, as well as the hydrolysis of the silane, which in turn forms microcracks and so leads to the decreased durability of the composite restoration (37). Indeed, microcracks or interfacial gaps at the intersection between the filler and the matrix permit stain infiltration and so facilitate discoloration (23,37).

In the formulation of Herculite XRV Ultra, TEGDMA was included to decrease the viscosity of the Bis-GMA resin. While the TEGDMA was included to enhance the handling features, it also resulted in water sorption ability (37). In fact, the hydrophilic characteristics of the TEGDMA lead to extraordinary water sorption, which prompts the extension and plasticizing of the Bis-GMA resin, and causes a reduction in the longevity of the composite resin. It has been reported that hydrophilic materials show a high degree of water absorption and, they become more colored with staining agents than hydrophobic materials (38).
Although Herculite XRV Ultra, a nano-hybrid composite resin, was expected to show less discoloration due to its small particle size, it showed a high degree of discoloration when immersed in mouthwashes. This can be attributed to the structure of the resin matrix (UDMA, TEGDMA, Bis-EMA) and the different sizes of the filler particles.

The results of this study showed that the color changes on composite specimens exposed to different types of mouthwash for 24 hours were not clinically acceptable (ΔE>3.3). Regarding the color parameters (L*a*b*), the results indicated that the ΔL values were negative, which showed that the composite samples became darker in all groups (except for the Chlorhex group). It was reported that color change for dimethacrylate based composites was mostly caused by ΔL* and Δb* (39). In terms of a* axis, the colors of the composite samples immersed in mouthwashes shifted from red towards green after SS application. In relation to the b* axis, the colors of the nanohybrid composite samples shifted from blue towards yellow after SS application (Table 3). Yellowing was most likely due to a reaction involving the many unreacted methacrylate groups (approximately 40%) present within the composite resin. The visual yellowing of BIS-GMA itself during UV illumination in the presence of oxygen implies that some chemical alteration of the resin itself may be one of the principal causes of the yellowing in dental composites (40).

Study Limitations

It is important to recognize the methodological limitations of in vitro studies designed to determine color stability. To accelerate the ageing process, in vitro studies aim to simulate the impacts of long-term exposure in an oral environment. Within the oral environment, the saliva and other liquids may dilute the stains. The first limitation of this study was not keeping the samples in artificial saliva as in the oral environment and not immersing them for a long time. Already, exposure of dental structures and restorative materials to the coloring agents in the oral environment is not continuous and the discoloration can be accelerated by mechanical wear. The second limitation of this study was that mechanical wear was not imitated. Besides, choosing only one composite resin and one surface sealant, and the lack of brushing effect with or without a toothpaste were the other limitations of the study.

Conclusion

Within the limitations of this study, it could be concluded that long-term use of mouthwash causes discoloration on a composite resin restoration. Besides, the application of a low viscosity liquid surface sealant material did not show the excepted effect on the color stability of a nano-hybrid composite resin in terms of three different mouthwashes.

Ethics

Ethics Committee Approval: Ethics committee approval is not required since our study was conducted with composite resin samples and no patient or dental samples were used.

Peer-review: Externally peer reviewed.

Authorship Contributions


Conflict of Interest: No conflict of interest was declared by the authors.

Financial Disclosure: The authors declared that this study received no financial support.

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